

Laid-Open Patent No.: 5-310495 (11/22/93)

Patent Application No.: 4-134411 (4/28/92)

Applicant: NKK

Inventors: ISHII, Shinji

OMURA, Masaki

NAKAHAMA, Yasumitsu

[Title of the Invention]

Process for silicon single crystal and manufacturing apparatus therefor

[Scope of the Claims]

[Claim 1]

A process for continuously drawing silicon single crystal, in which:

a rotatable quartz crucible containing fused silicon liquid is divided concentrically towards the center, using a plurality of partition members having through holes that cause said fused silicon liquid to flow through in one direction, into a raw material feeding zone, a fused liquid residence zone, and a crystal growth zone; and

the raw material is fed continuously in said raw material feeding zone and the fused silicon liquid, melted by a heating apparatus, resides for a prescribed residence period in said fused liquid residence zone, after which the fused silicon liquid is introduced via said through holes into the crystal growth zone, and silicon single crystal is drawn continuously in said crystal growth zone; characterized by the prescribed residence period of the fused silicon liquid in said fused liquid residence zone being 10 minutes or more and preferably 1 hour or more.

[Claim 2]

A process for silicon single crystal described in Claim 1, characterized by said plurality of partition members comprise an outer partition member having an ID [sic, inner radius?] r_o and an inner partition member having an ID [sic] r_i , and in the relationship between the ID [sic] r_c of the quartz crucible and the radius r_s of the drawn silicon single crystal, the members are arranged such that they fall within the range of the following conditions:

$$1) \frac{1}{4} (r_c - r_o) \leq (r_o - r_i) \leq 2 \cdot r_s$$

and

$$2) r_i > r_s + \alpha$$

where $\alpha \geq 1/2 r_s$

and

$$3) \text{substantially } (r_c - r_o) \geq 1 \text{ inch}$$

[Claim 3]

A process for silicon single crystal described in Claim 1, characterized by said plurality of partition members comprise an outer partition member having an ID [sic] r_o and an inner partition member having an ID [sic] r_i ,

and in the relationship between the ID [sic] r_c of the quartz crucible and the radius r_s of the drawn silicon single crystal, the members are arranged such that they fall within the range of the following conditions:

$$1) (r_c - r_o) \leq (r_o - r_i) \leq 2 \cdot r_s$$

and

$$2) r_i > r_s + \alpha$$

where $\alpha \geq 1/2 r_s$

and

$$3) \text{substantially } (r_c - r_o) \geq 1 \text{ inch}$$

[Claim 4]

A process for silicon single crystal described in Claim 1 in which, if in the outermost partition member of said plurality of partition members it is given that,

a circle having an area equivalent to the total area A_o of the through holes arranged on said member has an equivalent diameter d_o ;

the volume of fused liquid V grown into single crystal in a unit of time divided by said total area A_o is the equivalent through hole flow rate u_o ;

the fused liquid rotation speed at the ID [sic] r_o of said outermost partition member induced by the number of rotations n_c of said quartz crucible is u_y ;

and the terminal velocity of $10 \mu\text{m}$ air voids rising in the fused silicon liquid is u_b ;

then said equivalent through hole flow rate u_o is selected so that the relationship of the above is as follows:

$$100 \cdot u_b \leq V/(\pi d_o^2/4) \leq (1/100) \cdot u_y$$

[Claim 5]

A process for silicon single crystal described in Claim 1 in which, if in the innermost partition member of said plurality of partition members it is given that,

a circle having an area equivalent to the total area A_i of the through holes arranged on said member has an equivalent diameter d_i ;

the volume of fused liquid V grown into single crystal in a unit of time divided by said total area A_i is the equivalent through hole flow rate u_i ;

if the clockwise rotation direction as seen from above of the fused liquid in contact with the inner side of said innermost partition member is assumed to be positive, the absolute value of the difference between the product of the inner radius r_i of said partition member and the number of rotations n_c of the quartz crucible $r_i \times n_c$ and the product of the radius r_s of the single crystal and the number of rotations n_s of the single crystal $r_s \times n_c$ [sic, should be n_s] is the fused liquid rotation speed u_k of the single crystal growth zone;

and the terminal velocity of $10 \mu\text{m}$ air voids rising in the fused silicon liquid is u_b ;

then said equivalent through hole flow rate u_o is selected so that the relationship of the above is as follows:

$$100 \cdot u_b \leq V/(\pi d_o^2/4) \leq (1/200) \cdot u_k$$

[Claim 6]

An apparatus for manufacturing silicon single crystal having:

a rotatable quartz crucible containing fused silicon liquid;

a plurality of partition members that concentrically divide the interior of said quartz crucible in three locations or more and having through holes allowing the fused silicon liquid to flow toward the center in one direction;

a raw material feeding apparatus that continuously feeds the raw material in the outermost zone of the divided quartz crucible; and

a drawing apparatus that continuously draws silicon single crystal from the fused silicon liquid in the innermost zone of the divided quartz crucible;

characterized by configuring a fused liquid residence zone, between the outermost zone and the innermost zone of said divided quartz crucible, marked out by the outermost partition member and the innermost partition member, and by placing through holes on said outermost partition member and said innermost partition member such that the residence period of the fused silicon liquid in said fused liquid residence zone is 10 minutes or more, preferably 1 hour or more.

[Claim 7]

A process [sic, should be “an apparatus”] for manufacturing silicon single crystal described in Claim 6, characterized by said outermost partition member and the innermost partition member are each placed in a condition that satisfies the following conditions:

$$1) \frac{1}{4} (r_c - r_o) \leq (r_o - r_i) \leq 2 \cdot r_s$$

and

$$2) r_i > r_s + \alpha$$

where $\alpha \geq 1/2 r_s$

and

$$3) \text{substantially } (r_c - r_o) \geq 1 \text{ inch}$$

where r_s : radius of drawn silicon single crystal [cm]

r_c : inner radius of quartz crucible [cm]

r_o : inner radius of outermost partition member [cm]

r_i : inner radius of innermost partition member [cm]

[Claim 8]

A process [sic, should be “an apparatus”] for manufacturing silicon single crystal described in Claim 6, characterized by said outermost partition member and the innermost partition member are each placed in a condition that satisfies the following conditions:

$$1) (r_c - r_o) \leq (r_o - r_i) \leq 2 \cdot r_s$$

and

$$2) r_i > r_s + \alpha$$

where $\alpha \geq 1/2 r_s$

and

$$3) \text{substantially } (r_c - r_o) \geq 1 \text{ inch}$$

where r_s : radius of drawn silicon single crystal [cm]

r_c : inner radius of quartz crucible [cm]

r_o : inner radius of outermost partition member [cm]

r_i : inner radius of innermost partition member [cm]

[Claim 9]

An apparatus for manufacturing silicon single crystal described in Claim 6, characterized by the diameter d_A of the through holes placed on said outermost partition member is of a dimension satisfying the following:

$$100 \cdot u_b \leq V/(\pi n d_A^2/4) \leq (1/100) \cdot u_y$$

where d_A : diameter of through holes placed on the outermost partition member [cm]

u_b : terminal rising velocity of 10 μ m OD air voids rising in the fused silicon liquid [cm/sec]

V : volume of fused liquid grown into single crystal in a unit of time [cm³/sec]

n : number of through holes on the outermost partition member

u_y : fused liquid rotation speed, which is the product of the inner radius r_o of the outermost partition member and number of rotations n_c per unit of time of the quartz crucible [cm/sec]

[Claim 10]

An apparatus for manufacturing silicon single crystal described in Claim 6, characterized by the diameter d_B of the through holes placed on said innermost partition member is of a dimension satisfying the following:

$$100 \cdot u_b \leq V/(\pi n d_B^2/4) \leq (1/200) \cdot u_k$$

where d_B : diameter of through holes placed on the innermost partition member [cm]

n : number of through holes on the innermost partition member

u_b : terminal rising velocity of 10 μ m OD air voids rising in the fused silicon liquid [cm/sec]

V : volume of fused liquid grown into single crystal in a unit of time [cm³/sec]

u_k : crystal growth zone maximum rotation speed, which is the difference between the product of the inner radius r_i of said partition member and the number of rotations n_c of the quartz crucible $r_i \cdot n_c$ and the product of the radius r_s of the single crystal and the number of rotations n_s of the single crystal $r_s \cdot n_s$, expressed as an absolute value [cm/sec].

[Claim 11]

An apparatus for manufacturing silicon single crystal described in Claim 6, characterized by the through holes of said innermost partition member are placed in positions where a portion of the fused silicon liquid flowing through said through holes forms a free surface of fused silicon liquid.

[Claim 12]

An apparatus for manufacturing silicon single crystal described in Claim 6, characterized by the minimum angle α of the through holes placed on said outermost partition member and the through holes placed on the innermost partition member as seen from the central rotation axis of the quartz crucible is $90^\circ < \alpha \leq 180^\circ$.

[Claim 13]

An apparatus for manufacturing silicon single crystal described in Claim 6, characterized by the minimum

angle α of the through holes placed on said outermost partition member and the through holes placed on the innermost partition member as seen from the central rotation axis of the quartz crucible is $120^\circ < \alpha \leq 180^\circ$.